SULFATE AND ACID RESISTANCE OF MATERIALS FOR USE IN SEWAGE AND WASTEWATER TRANSPORT SYSTEMS IN SAUDI ARABIA - A REVIEW

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SUMMARY: Concrete and metallic structures in sewer transport systems in the Eastern Province of Saudi Arabia showed signs of severe deterioration within 3 years into service. Several investigations were carried out to determine the corrosivity of the sewer environment and severity of material degradation in the sewer systems. At the same time studies were conducted to find corrosion resistant materials and coating suitable for use in sewer environments. In this paper a review of the findings of these investigations is given.

Several different materials, such as high performance coatings, fiber glass reinforced linings, special mortars, brick or ceramic linings, etc., ar used to protect concrete from sulfuric acid attack in a sewage environment. Two proprietary high alumina cementitious lining materials, SC and CC, are recent additions to the list of protective materials used in the Arabia Gulf. This report documents the findings of a laboratory study under accelerated conditions in addition to a two-year field study in a wastewater pump station in Jubail, Saudi Arabia.

Performance of the liner materials at the wastewater pump station was monitored for sulfate content and alkalinity after 6, 12, and 24 months of exposure. The analysis and evaluation test data generated from the accelerated lab study and the field study showed that SC performed, superior to CC within the test conditions used in this investigation.

KEYWORDS: cementitious, concrete durability, corrosion, biogenic, fly ash, high alumina cement, Saudi Arabia, sewer, silica fume, sulfate resistance, sulfuric acid, wastewater.

INTRODUCTION

The problem of biologically induced corrosion on sewer structures in the Middle East is serious and more severe than that encountered in other parts of the world. This is mainly due to the high effluent temperature caused by the hot climate, shortage of water

H. Saricimen. Sulfate resistant materials research for use in wastewater transport systems in Saudi Arabia - a review. Calcium Aluminate Cements: Proceedings of the Centenary Conference. Avignon. France, 31 June - 2 *July 2008.* iHS BRE Press, 2008.

for frequent flushing, the high level of natural sulfates in water, and low flow velocity of wastewater resulting from topographical flatness on the ground [1-3, 8].

Severe sulfate attack has been reported on the concrete walls of pump stations and transport systems in the Eastern Province of Saudi Arabia within 3 years after taking various systems into service [1-3]. Investigations showed that structures above liquid level, particularly in one of the stations, were badly corroded. Coal-tar epoxy coatings on the concrete walls were completely deteriorated on the unsubmerged parts of the walls and along the splash zone (liquid level). The concrete was also deteriorated, to a depth of 1–3 cm, on all the unsubmerged surfaces with a mushy appearance and white colour, Figure 1. Sulfate profiles in terms of $\%SO_3$ of the core samples retrieved from the four walls of the lift station showed that sulphate content in 0 – 3.8 cm thickness of the cores ranged from 6.11 to 92.6% by weight of cement. These values are far above the allowable sulphate concentration (3% by weight of cement according to BS8110 Part-1) indicating nearly all the cement in the 3.8 cm thick surface layer of the walls deteriorated by sulphate attack [1].

Fig. 1: *Corrosion in wastewater pump station.*

The coating on the submerged parts of the walls and the concrete appeared to be intact and in good condition without any sign of deterioration. The only exception was blistering of coal tar epoxy coating at the bottom surface of the concrete pump station [1]. Likewise, the metallic structures above liquid level had their coal tar epoxy or zinc coatings failed and the metal substrate underwent generalized pitting.

The submerged parts of the metallic structures and coatings on them were intact with no sign of severe deterioration or corrosion [1, 3].

The in-situ investigation showed that corrosion observed on the concrete and metallic surfaces in the pump stations was caused by sulfuric acid attack on the unsubmerged (above liquid level) of the structures [1-3].

To evaluate corrosivity of environment in these pump stations several studies were undertaken. Rectangular shaped metallic specimens (corrosion coupons), coated with several brands of polymeric corrosion resistant coatings were prepared and exposed to the above liquid level environment in one of the pump stations for several months. The coupons were retrieved and evaluated in the laboratory for corrosion resistance of the coatings [4].

The studies also included investigation of corrosion resistance of high alumina cement based lining materials designated by CC and SC in the laboratory and in sewer environment on concrete surfaces of one of the pump stations [5, 6].

This paper gives a review of the earlier investigations pertinent to sewer corrosion in the middle East and documents our investigations of corrosion resistant materials including proprietary CC and SC in sewer environments at Jubail, in the Eastern Province of Saudi Arabia [1, 6].

THEORETICAL BACKGROUND OF SEWER CORROSION

In early decades of the twentieth century, concrete technologists showed that low tricalcium aluminate, $3CaO.Al_2O_3$ (C3A), cement reduced concrete deterioration caused by sulfate attack [20]. It was reported later that sulfate attack appears in different form in sewer structures [7, 8]. Sewer structures are subjected to sulfuric acid attack. Sulfuric acid is formed on aerobic surfaces by oxidation of hydrogen sulfide (H_2S) in the presence of humidity (H_2O) and aerobic bacteria *Thiobacilli* [9-11].

Hydrogen sulfide corrosion of sewer systems was known as early as 1902 [10]. It was, however, thought to be a pure chemical reaction between hydrogen sulfide and sewer structures. Later research showed that sulfide corrosion involves a combination of bacteriological as well as chemical processes resulting in sulfuric acid attack on concrete and metallic surfaces in the sewer systems [12-15].

Domestic wastewater is usually composed of 99% water. The total dissolved, suspended, and settleable solids account for 1400 mg/L at the highest. The wastewater contains about 20mg/L of inorganic sulfate and 6mg/L of organic sulfur compounds. The pH varies generally between 6.5 and 7.5. Although the wastewater may not appear to be corrosive liquid at the beginning, the great variety of micro-organisms in it break down the organic matter, sulfur and sulfate compounds, into NH_3, CO_2, and H_2S and makes the liquid slightly acidic [9, 10, 12, 13]. In the acidic wastewater, the H_2S is released into sewer atmosphere. The released hydrogen sulfide is then absorbed into moisture condensed on the concrete walls and metal surfaces in the sewer. The absorbed hydrogen sulfide is readily oxidized in the presence of moisture (H_2O) and aerobic bacteria *Thiobacilli*. The concentration of sulfuric acid in the condensed water film on the sewer surfaces may exceed 5% and the pH of the water film may be as acidic as pH = 1, even when the pH of wastewater stream is 6–7 [1, 8, 9-18]. Consequently the aerobic environment becomes severely corrosive to all structures in the system.

Aerobic corrosion of metallic and concrete structures in sewers

The structures in the aerobic part of the sewers in presence of free oxygen are exposed to sulfuric acid corrosion as explained above. Metallic structures lose their coatings, coal tar epoxy or zinc, and undergo severe generalized pitting.

One of the common forms of sulfate attack on concrete takes place with calcium hydroxide, $Ca(OH)_2$, to form gypsum, calcium sulfate $(CaSO_4).2H_2O$ As a consequence, the pH decreases. Gypsum, being an expansive compound, leads to increase in volume and causes softening of the concrete and loss of strength [19].

There are other common sulfate reactions which take place on the hydration products of alumina-bearing compounds in cement. In the presence of calcium hydroxide, sulfate ions react with tricalcium aluminate, $3CaO.Al_2O_3$ (C_3A), and form ettringite, $3CaO.Al_2O_3.3CaSO_4.31H_2O$. Ettringite formation also results in expansion, loss of strength and mass in the concrete [20, 21].

Gypsum may also react with calcium carbonate and with calcium silicate hydrates, C-S-H, in cement to form thaumasite, $CaSiO_3.CaSO_4.CaCO_3.15H_2O$. Thaumasite transforms hardened concrete to a pulpy mass and causes severe damage [20].

Anaerobic corrosion in sewer systems

Direct reaction of hydrogen sulfide (H_2S) with the sewer structures is the most common form of anaerobic corrosion observed in the sewer systems. In this, hydrogen sulfide

reacts with iron, in an aqueous environment in the absence of free oxygen, to form iron sulfide according to the following reaction:

$$Fe + H_2S \rightarrow FeS + H_2$$

At pH values of 7 and above (favourable pH for growth of *Desulfovibrio*), ferrous sulfide (FeS) precipitates on areas close to anodic sites on the structure and forms a protective film [12]. Upon prolonged exposure to the environment and bacterial cultures, the sulfide film becomes detached from the metal surface, causing formation of FeS/Fe couple and an increase in the corrosion rate.

In addition, there appears to be other mechanisms that contribute significantly to corrosion of metals in anaeroboic environments. Micro-organisms, for example, can develop oxygen or ion concentration cells on metal surfaces as a result of their active metabolic processes. Metal goes into solution at the anodic areas of low oxygen concentration under the mass of micro-organisms. It was shown by Von Wolzogen Kuhr and van der Vlug [22] that sulfate reducing bacteria (SRB) which contain the molecular-hydrogen-activating enzyme hydrogenase remove hydrogen from iron surface (cathodic depolarization) and utilize it for the reduction of sulfate. As electrons are removed, more iron goes into solution. The presence of phosphide (Fe_2P) was reported [23] in the corrosion product of mild steel under anaerobic conditions. During anaerobic corrosion, highly volatile phosphorous compound reacts with iron to form Fe_2P. It was shown in laboratory that a non-marine strain of *Desulfovibrio* produced a volatile organophosphorous compound which reacts with mild steel to form iron phosphide (Fe_2P) [23]. The corrosion product is an amorphous type of iron phosphide which can be detected by the formation of phosphine upon its acidification. Phosphine (in addition to H_2S) has been detected from all the cases of suspected anaerobic corrosion (including tubercules from the inside of water pipes) examined so far [23].

EXPERIMENTAL WORK AND RESULTS

1. Evaluation of pump stations, corrosion of concrete and metallic structures

The investigation of the concrete structure in pump stations involved half-cell potential measurement of reinforcing steel bars using a high impedance voltmeter and a Cu-$CuSO_4$ (CSE) standard electrode. The temperature of the wastewater was monitored over the period of investigation. The pH of the condensed moisture on the sewer structures was measured using narrow-range pH indicating papers. Relative humidity in the pump stations was recorded using a recording hygrometer located about 2 metres below the top of the manhole cover [1-3, 24].

Wastewater samples taken from the pump station were analyzed for pH, biological oxygen demand (BOD), chemical oxygen demand (COD), sulfide, sulfate, chloride, total solids, oil and grease, dissolved oxygen, and bacteriological count. The results of these and other tests are summarised below.

The wastewater analysis showed that it was weakly acidic with pH between 5.1 and 6.8. The BOD ranged from 150 to 580 mg/L, and the COD varied between 361 to 830 mg/L in the wastewater. The bacterial count was in the range of 2.6×10^6 to 140×10^6 per ml indicating high bacterial activity. The hydrogen sulfide content varied from 0.4 to 4.8 mg/l, indicating substantial amount of dissolved H_2S in the wastewater. The

chloride and sulfate contents, on the other hand, were low. The wastewater temperature ranged between 31 and 34°C. The pH of condensed moisture was very acidic and about 1.0. The relative humidity in the pump station was 100% most of the time.

Cylindrical concrete core samples of 75×200 mm were retrieved from the walls of the pump station and tested in the laboratory for pulse velocity, water absorption, electrical resistivity, and chloride and sulfate concentration. The sulfate and chloride profiles of the core samples were determined at 0.5, 7.5 and 15 cm depth from the top surface using gravimetric and titration methods, respectively. Energy dispersive X-ray analysis of the cores was also carried out.

The half-cell potentials of the reinforcements in the four walls of the pump station were in the range of -250 mV vs CSE near the top of the wall to -590mV vs CSE near the bottom of the wall indicating active corrosion of reinforcements at the bottom. The concrete cover thickness varied from 38 to 55 mm on the interior face of the walls and 60 to 65mm on the exterior face of the walls.

The non-destructive testing of concrete properties showed that the pulse velocity varied in the range of 3,300 to 3,900 m/s. The 30 minute absorption values were in the range 3.5 to .6%. The electrical resistivity ranged from 2,400 to 12,000 Ohm-cm. The chloride concentration varied in the range of 0.10 to 0.60% by weight of concrete. The sulfate concentration was in the range of 0.8 to 13.04% by weight of concrete. The sulfate concentration was high at the surface (inside) and decreased sharply with depth.

Elemental and compound analysis of deteriorated concrete by energy dispersive X-ray analysis showed that the deteriorated concrete was mainly composed of silicon dioxide (SiO_2) and gypsum ($CaSO_4.2H_2O$).

The investigation showed that concrete walls deteriorated, in a very short period after being taken into service (about 3 years), up to 3 cm depth by sulfuric acid attack causing formation of gypsum in the deteriorated layer. The reinforcements did not indicate any corrosion and concrete quality was on the other hand in good condition. Wastewater analysis confirmed that the environment is loaded with bacteria and fit for the growth of anaerobic bacteria and generation of hydrogen sulfide. Consequently, the aerobic surfaces are subjected to severe sulfuric acid corrosion [1, 2, 24].

2. Evaluation of corrosion resistant coating systems for sewer structures

In this study performance of structural materials and coating systems exposed to aerobic sewer environment was investigated [4].

Uncoated coupons of size 50×75 mm were prepared from galvanized steel (GS), ductile iron (DI), polyvinyl chloride (PVC), AISI 316 stainless steel, aluminum, brass and glass fiber reinforced plastic (GFRP). After initial weighing they were installed in one of the pump stations in triplicate in a wooden rack, and exposed to aerobic environment for 87 days before retrieving for analysis in the laboratory.

For coated coupons, mild steel plates of 50×75 mm were coated with eight different high quality polymeric coating systems; two kinds of vinylester, high performance coal tar epoxy, high thickness micaceous iron primer based on chlorinated rubber, polyamide cured epoxy, amine adduct cured coal-tar epoxy, amine cured epoxy resins, and polyamide cured epoxy coating, provided by three internationally known paint manufacturers. The coated coupons were then installed in triplicates on wooden racks in the lift station. They were retrieved after 490 days of exposure to the aerobic environment in the pump station and evaluated.

The evaluation consisted of visual inspection, analysis of coupons (metallic and coated) by gravimetric and metallographic techniques, and electrochemical measurements [1, 25, 26].

The results showed that the AISI 316 Stainless steel, PVC and GFRP performed well, whereas the ductile iron, galvanized steel, brass and aluminum were demonstrated to be unsuitable as structural materials in aerobic environment in the pump station without adequate protection. Ductile iron and galvanized steel corroded by generalized pitting, brass corroded by dezincification (de-alloying) and pitting, and aluminum corroded by crevice corrosion. The SEM and EDX investigations showed that the corrosive species were sulfur containing compounds, most probably H_2S and H_2SO_4. Chloride ions might have also contributed to the corrosion of metallic coupons. Chloride ions are strong depassivating agents for iron base alloys. They react with iron to produce chlorides and ultimately oxides of iron. Although presence of phosphorous was shown in the corrosion products of iron based alloys in this study, no evidence for Fe_2P was observed.

The coated coupons were first rated according to their visual appearance. Two of the coated coupons were eliminated from further analysis due to severe deterioration of the coatings. The remaining six coated coupons were subjected to electrochemical testing. "Potential vs Time" and "Resistance vs Time" curves were generated for the six coated coupons. The results of visual and electrochemical evaluation on the basis of point system showed that the coatings rated in the following order according to their corrosion resistance in aerobic environment in the pump station:

1st (Best)	Vinyl ester (Manufacturer A)
2nd	Polyamide cured epoxy
3rd	Amine cured epoxy resin and Polyamide cured epoxy coating
4th	Vinyl ester (Manufacturer B)
5th	Amine adduct cured coal tar epoxy

3. Evaluation of high alumina cementitious materials for use in sewer systems

Two cementitious lining materials, 'CC' and 'SC', are recent additions in Saudi Arabia, to the list of protective materials in sewer and sulfuric acid environments. They are both high alumina proprietary products. According to the manufacturer's catalogue, CC is high alumina cement based on calcium aluminates and developed for resistance to acid and biogenic corrosion. SC is a ready to use product consisting of corrosion resistant calcium aluminum cement and specially synthesized calcium aluminate aggregates. The product is designed for use in rehabilitation of sewage structures. Both of the products and special coarse sand which was mixed with the coating materials were obtained from their supplier Raymond S.A. Ltd in Saudi Arabia. This section of the paper documents the findings of a laboratory study under accelerated conditions as well as the findings of a two-year field study in a wastewater pump station in Jubail, Saudi Arabia [5, 6].

For laboratory investigation, mortar cubes of 50 mm size were prepared using special silica sand and (1) Type-I PC +8% silica fume (SF), (2) Type-I PC +20% fly ash (FA), (3) Type-I PC, (4) CC and (5) SC. The cube specimens were cured for 7 days under wet burlap, followed by 21 days air curing in the laboratory conditions. In the cube specimens, a sand-to-cementitious material ratio (s/c) of 2.0 and an effective water-to-cementitious material ratio (w/c) of 0.45 were used. After curing, the cubes for sulfate and alkalinity testing were coated with wax on all surfaces except one surface to facilitate a unidirectional exposure to sulfate ions. The cubes were immersed in a 2%

sulfuric acid solution in a Plexiglas container. The container was kept covered throughout the testing period. The pH and concentration of the acid solution were monitored and the concentration was adjusted periodically. Distilled water was added to the acid bath to compensate for the loss of water by evaporation.

First group of cube specimens were retrieved after 73 days of exposure to sulfuric acid. They were washed with potable water, air dried and tested for compressive strength. Diluted water extracts of the powdered mortar samples were tested for alkalinity and sulfate content [27, 28]. The alkalinity was assessed by measuring pH by using a digital Orion ion analyzer. The remaining cubes ere kept for another 77 days (total of 150 days exposure) in sulfuric acid solution before testing.

For the field study, walls and the ceiling of the pump stations in Jubail were sand blasted and coated with proprietary high alumina CC and SC mortars at a thickness of 15 mm according to the supplier's instructions by their agent in the Kingdom [1]. The mortar coatings were exposed to actual wastewater environment in the pump station for two years. The pump station was inspected after 0, 6, 12 and 24 months of exposure of the coatings to the wastewater environment. At each inspection, the appearance of the coating on the walls and the ceiling was noted. Powder samples were taken from the walls and ceiling at 5, 10, and 15 mm depths from the coatings and at 10 and 20 mm depths in the concrete underneath. The powder samples were tested for alkalinity and sulfate content. For each data point in all the analysis at least duplicate samples were tested. Compressive strength measurements were determined by testing the 50 mm specimens in triplicate.

3.1 Laboratory test results

Laboratory test results are summarized in Table 1. There was not much change in physical appearance of the mortar cube specimens immersed in 2% sulfuric acid solution after 25 days. After 150 days, however, the SC mortar cubes were in the best condition and the SF containing mortar specimens in the worst.

The weight change was the least in SC mortar cube specimens. It was 0 and 0.56% after 73 and 150 days immersion in the sulfuric acid solution, respectively. The weight change was the highest in 8% SF containing mortar specimen. It was 23 and 79 % after 73 and 150 days immersion in the acid solution, respectively.

The compressive strength reduction in SC mortar specimens was the least. It was 17 and 34 % after 73 and 150 days immersion in the acid solution, respectively. The compressive strength reduction also was the highest in SF containing specimens. It was 34 and 78 % after 73 and 150 days of exposure to the acid solution, respectively.

The SC mortar cubes exhibited the lowest sulfate penetration after both immersion periods. Sulfate concentration decreased with depth from the surface of the cubes and increased with duration of immersion. The sulfate concentration in SC mortar cubes was 0.37 and 3.77% by weight of mortar, in 0-10mm depth after 73 and 150 days, respectively.

The alkalinity, pH, was the highest in the 0-10 mm depth of SC mortar cubes. The pH decreased from 10.9 to 8.9, in 0-10 mm thickness of the SC mortar cubs after 150 days immersion in 2% sulfuric acid solution.

Table 1. *Laboratory test results.*

Properties/ Specimens		SC		CC		SF		FA		Type I	
		73 d	150 d	73 d	150 d	73 d	150 d	73 d	150 d	73 d	150 d
Visual (after 150 days)		EX	**G**	VG	**G**	VG	**VB**	VG	**B**	VG	**F**
Weight change (%)		0.0	**0.56**	13	**43**	23	**79**	18	**69**	17	**67**
Compressive strength reduction (%)		17	**34**	32	**48**	34	**78**	34	**74**	15	**72**
Sulfate content (%)	0-10 mm depth	0.37	**3.77**	0.52	**3.19**	1.96	**3.41**	1.76	**3.19**	0.81	**3.53**
	40-50 mm depth	0.13	**0.04**	0.12	**0.13**	0.09	**0.23**	0.09	**0.07**	0.10	**0.13**
Alkalinity (pH)	0-10 mm depth	10.98	**8.95**	9.50	**8.50**	11.94	**4.20**	11.97	**4.20**	12.02	**4.10**
	40-50 mm depth	11.26	**11.20**	10.17	**10.29**	11.96	**11.80**	11.92	**11.77**	11.94	**11.85**

EX – Excellent; VG – Very Good; G – Good; F – Fair; B – Bad; VB – Very Bad

3.2 Field test results

Some of the field test results are summarized in Figures 2 and 3. During two-year exposure, no change was noticed in the physical appearance of the walls of the wastewater pump station.

Sulfate profile in the SC and CC mortar coating on the upper part (aerobic) of the short walls of the pump station is shown in Figure 2. The CC mortar coating has lower sulfate profile in the pump station compared to SC mortar coating. The sulfate content in both the mortar coatings increased with duration and decreased with depth as expected.

The pH profile variation in SC and CC mortar coatings in the pump station is shown in Figure 3. The pH of the coatings decreased with exposure period. The results showed that the SC mortar performed better than the CC mortar.

Overall evaluation of the laboratory and field results showed that SC mortar performed better than CC mortar in a sulfate environment. In the laboratory, performance of the materials tested in 2% sulfuric acid solution for 150 days ranked in the order of:

SC>CC>Type-I+20%FA>Type-I>Type-I +8%SF.

CC>SC in terms of sulfate content, and SC>CC in terms of alkalinity.

In the field, however, after 24 months of exposure in a wastewater pump station, performance of the two proprietary lining materials SC and CC was not very clear:

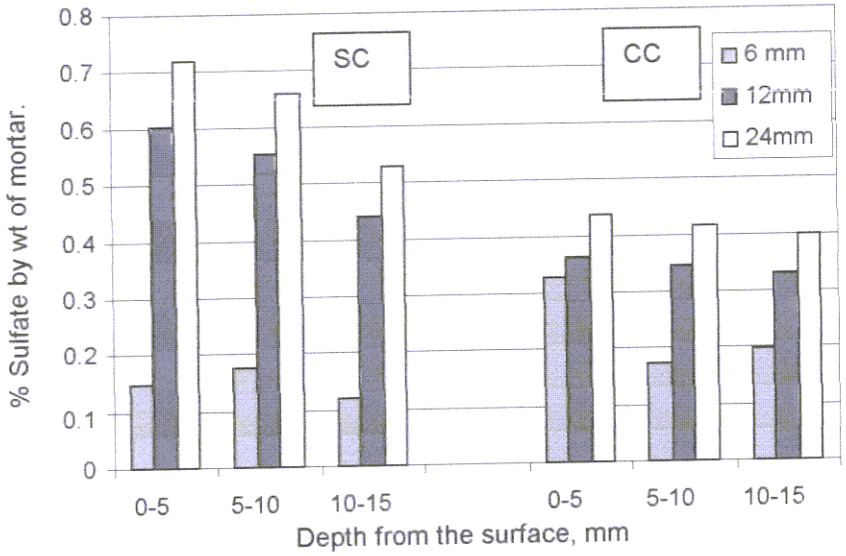

Fig. 2 *Sulfate profile in the mortar cover.*

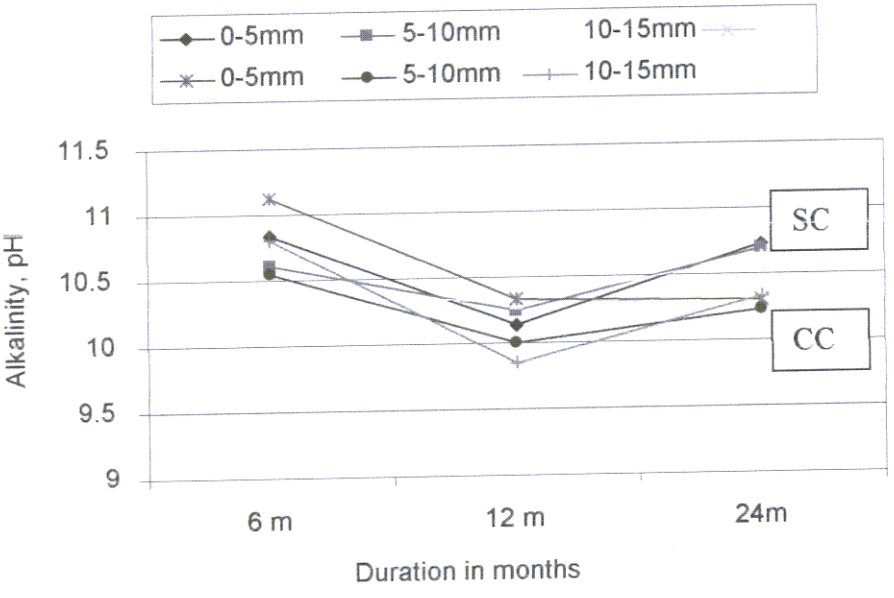

Fig. 3 *Alkalinity variation in the mortars with depth and time.*

CONCLUSIONS

1. The domestic wastewater in the pump stations in Jubail, Saudi Arabia, was weakly acidic (pH = 5.1– 6.8), and moderately corrosive to submerged metallic and concrete structures. It was, however, a potential environment for hydrogen sulfide generation due to the presence of large number of anaerobic bacteria, most probably *Desulfovibrio*, and lack of dissolved oxygen. The condensed moisture on the submerged structures was

highly acidic due probably to conversion of H_2S into sulfuric acid on moist sewer surfaces in the presence of aerobic bacteria *Thiobacilli.*

2. Concrete deterioration on the unsubmerged walls (aerobic environment) was very severe. The coal tar epoxy coating on them deteriorated and gypsum formation occurred due to sulfuric acid attack on the surface layer of the walls to a depth of about 3 cm. Metal structures such as DI and GS also corroded severely in the aerobic environment by generalized pitting.

3. Coupon study to find most corrosion resistant materials and coatings suitable for sewer environment showed that:

(a) Stainless steel was the most corrosion resistant material in this environment. The PVC and GFRP coupons were also unaffected in the sewer environment. Corrosion of DI coupons progressed with generalized pitting which was faster at locations of high graphite/iron ratio. The corrosion of GS coupons proceeded by depletion and cracking of the zinc layer on the coupons. Aluminum coupons showed crevice corrosion under sludge deposits. Brass coupons corroded by dezincification.

(b) Regarding performance of polymeric coatings, vinylester and polyamine cured epoxy coatings were the best performers of the eight different coatings tested in the sewer environment.

4. The laboratory tests of proprietary high alumina cement, CC and SC mortar cubes in comparison with mortar cube specimens containing 8%SF+Type-I, 20%FA+Type-I, and Type-I cement showed that SC performed the best in 2% sulfuric acid solution.

In the pump station, however, superior performance of SC mortar coating in sewer environment where actual corrosive agent is bionic sulfuric acid, was not so distinct compared to the performance of CC mortar after two years exposure. The CC mortar performed better in terms of sulfate penetration whereas the SC mortar showed better performance in terms of alkalinity profile and pH.

5. Therefore, it is recommended that the field investigation should be pursued beyond two years period for long term durability evaluation of the tested high alumina cements, SC and CC, because sulphate attack in a wastewater manhole is a slow process.

ACKNOWLEDGMENT

The author wishes to acknowledge that the research reported in this paper were carried out at the KFUPM Research Institute, Center for Engineering Research and sponsored by and published with the permission of the Directorate General for Jubail Project, Royal Commission for Jubail and Yanbu.

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